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The work sponsored under this contract has centered on the molecular beam epitaxial growth of InSb/CdTe heterojunctions and multilayer structures, and on (Zn,Cd)Te/CdTe strained layer superlattices. We have been the first to demonstrate the molecular beam epitaxial growth of InSb/CdTe superlattices at temperatures necessary for the growth of electrical active InSb. An indication of the progress in this III-V/II-VI mixed system is the first observation of the quantum hall effect (QHE) in the InSb/CdTe heterojunction, by Simon Greene at the Cavendish Laboratory. As a result of the demonstration of a 2DEG and the ability to grow InSb/CdTe superlattices, the realization of infrared lasers and detectors in the 3-5 μ m region has been significantly enhanced. We have reported the growth of a novel semiconducting material In₂Te₃. Work on the II-VI material compounds has centered on the growth of (Zn,Cd)Te/CdTe strained layer superlattices that have the layer thickness ratios and Zn composition tailored to allow the in-plane lattice parameter match that of Hg_{0.8}Cd_{0.2}Te.

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INTRODUCTION

During the past twelve months, the work sponsored under this contract has centered on the molecular beam epitaxial growth of InSb/CdTe heterojunctions and multilayer structures, and on (Zn,Cd)Te/CdTe strained layer superlattices. In both these systems the work at the Night Vision Laboratory under this contract has been recognized as one of the major innovators in this field of research. We have been the first to demonstrate the molecular beam epitaxial growth of InSb/CdTe superlattices at temperatures necessary for the growth of electrical active InSb. An indication of the progress in this III-V/II-VI mixed system is the first observation of the quantum hall effect (QHE) in the InSb/CdTe heterojunction, by Simon Greene at the Cavendish Laboratory. As a result of the demonstration of a 2DEG and the ability to grow InSb/CdTe superlattices, the realization of infrared lasers and detectors in the 3-5 μ m region has been significantly enhanced. We have reported the growth of a novel semiconducting material In₂Te₃. Work on the II-VI material compounds has centered on the growth of (Zn,Cd)Te/CdTe strained layer superlattices that have the layer thickness ratios and Zn composition tailored to allow the in-plane lattice parameter match that of Hg_{0.8}Cd_{0.2}Te.

InSb/CdTe

We have shown that significant improvements in interfacial quality of the InSb/CdTe heterojunction can be obtained by the proper choice of fluxes during growth. The use of a Cd/Te flux ratio of 3:1 ($J_{Cd}/J_{Te}=3$) during the growth of CdTe has enabled epitaxy at a substrate temperature of 300°C. Interfaces formed with this flux ratio are abrupt, in sharp contrast to those formed under stoichiometric flux conditions ($J_{Cd}/J_{Te}=1$). Subsequent growth of InSb at a substrate temperature of 300°C on thin CdTe epilayers (400 and 800 Å) has been examined as a function of the InSb growth rate and Sb/In flux ratio. The quality of the interfaces shows a progressive improvement with increasing InSb growth rate. The Cd enhanced flux during the growth of the CdTe layers has enabled epitaxy of InSb/CdTe superlattices. The superlattices have been grown with a range of period thicknesses from 200Å to 1800Å and the resulting structures have been characterized by cross-sectional TEM and X-ray diffraction. Dynamical X-ray diffraction theory has been applied to the structures and has been compared with the experimental spectra. We have shown that excellent structural properties can be obtained for period thicknesses greater than 450Å.

In_2Te_3

In the course of our studies into the molecular beam epitaxial growth of InSb/CdTe multilayers and superlattices, we found that there was a strong chemical reaction at the InSb/CdTe interface during growth at substrate temperatures compatible with the growth of high structural and electronic quality of both InSb and CdTe (275-300 °C). Recent studies of samples grown at the Night Vision Laboratory by Raman spectroscopy conducted by Dietz Zahn at Aachen, Germany of the heterojunction formed from the growth of CdTe on InSb under stoichiometric flux conditions and growth temperatures of 200-300°C revealed that the InSb and CdTe react to form a complex interface identified to consist predominantly of In_2Te_3 and elemental Sb. In_2Te_3 is a defect zinc blende type semiconductor with a bandgap of approximately 1 eV, lying between that of InSb ($E_g=0.18\text{eV}$) and CdTe ($E_g=1.44\text{eV}$). Thermodynamic considerations of the stability of In_2Te_3 with respect to InSb and CdTe led us to examine the possibility of MBE growth of In_2Te_3 for use as a barrier between the CdTe and InSb to facilitate fabrication of InSb/CdTe multilayers, and the interesting possibility of exploiting the large difference in badgaps between InSb and In_2Te_3 to grow InSb/ In_2Te_3 multilayers for novel quantum well structures. In addition the unique structure of In_2Te_3 , with 1/3 of the In sublattice sites vacant, is of fundamental interest for molecular beam epitaxial growth dynamics. We have shown that thin film (500Å-7000Å) single crystal In_2Te_3 can be grown successfully on

InSb(100) homoepitaxial layers at substrate temperatures of 300 - 350°C and Te/In flux ratios of 3/2 - 5/2. Epitaxy was monitored by reflection high energy electron diffraction and the stoichiometry of the grown layers assessed by Auger spectroscopy and energy dispersive X-ray analysis. Raman studies of the layers have been conducted and compared with a bulk In_2Te_3 standard. The crystal structure has been determined by X-ray diffraction using Weissenburg and oscillation photographs, confirming that the layers have a f.c.c. crystal structure with a lattice parameter of 18.50Å, in excellent agreement with the bulk value. Bandgap measurements have been performed on the layers by photoreflectance. Molecular beam epitaxial growth of InSb and CdTe on epitaxial In_2Te_3 films for fabrication of InSb/ In_2Te_3 /InSb and InSb/ In_2Te_3 /CdTe multilayers has been studied. Auger depth profiling of the resulting layers shows severe intermixing into the In_2Te_3 .

(Zn,Cd)Te/CdTe strained layer superlattices

The need for a suitable low defect density, lattice matched substrate or substrate/buffer combination for the molecular beam epitaxial growth of thin film (Hg,Cd)Te led us to examine the use of (Zn,Cd)Te/CdTe strained layer superlattices. (Zn,Cd)Te/CdTe strained layer superlattices have been grown by molecular beam epitaxy on CdTe(100), InSb(100) and GaAs(100) substrates and substrate/buffer combinations with period thicknesses ranging

from 90Å to 330Å and with total superlattice thicknesses of 0.8µm and 1.6µm. The ratio in thickness between the (Zn,Cd)Te and CdTe strained layers has been tailored to allow the in plane lattice parameter of the free standing superlattice match the lattice parameter of $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$. The resulting structures have been characterized by X-ray diffraction techniques. Multiple satellite peaks have been observed indicating that these superlattices can be grown with a high structural quality. The structural quality has been found to be independent of the superlattice period but dependent on the originating substrate.

APPENDIX OF PAPERS PUBLISHED OR TO BE PUBLISHED

Molecular Beam Epitaxial growth of InSb/CdTe heterojunctions for multilayer structures.

T.D. Golding, M.Martinka, and J.H.Dinan.

J. Appl. Phys. **64** (4), 1873 (1988)

Molecular Beam Epitaxial growth and Characterization of In_2Te_3 .

T.D.Golding, P.R.Boyd, M. Martinka, P.M. Amirtharaj,J.H. Dinan,S.B.

Qadri

D.R.T. Zahn and C.R.Whitehouse.

(JAP submitted November 1988)

MBE growth and X-ray Characterization of (Zn,Cd)Te/CdTe Strained Layer Superlattices.

T.D. Golding, S.B. Qadri, and J.H. Dinan.

(JVST submitted October 1988)

InSb/CdTe superlattices grown by molecular beam epitaxy.

T.D. Golding, J.H.Dinan A.G. Cullis, G.M. Williams, S.Barnet,

C.R.Whitehouse

J.E.Macdonald and K.M.Conway.

(JAP to be published)

Magnetoresistance of quasi-2DEG in InSb/CdTe heterostructures.

S.K.Greene, T.D.Golding, M.Pepper and J.H.Dinan.

(SMS submitted June 1988)

Quantum Hall Effect in two-dimensional electron gas in InSb/CdTe heterojunction.

S.K.Greene, T.D.Golding, M.Pepper and J.H.Dinan.

(to be published)

Raman Spectroscopy of InSb/CdTe heterojunction: Improved Interface Quality obtained by Cd-Overpressure during Molecular Beam Epitaxial Growth.

D.R.T. Zahn, T.D. Golding , K.J. Mackey , J. Geurts J.H. Dinan , W.

Richter , and R.H. Williams.

(JAP submitted June 1988)

The Influence of Cd-Overpressure in Molecular Beam Epitaxy of InSb/CdTe Heterostructures: A combined Raman and Infra-Red Spectroscopy Study.

D.R.T. Zahn, T.D. Golding, K.J. Mackey, J. Geurts J.H. Dinan, W. Richter, and R.H. Williams.

(JAP submitted October 1988)

Anisotropic Defect Distribution in CdTe/(Cd,Zn)Te Strained Layer Superlattices.

P.D. Brown, T.D. Golding, G.J. Russell J. Woods and J.H. Dinan.

(to be published)

Molecular-beam epitaxial growth of InSb/CdTe heterojunctions for multilayer structures

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We have used the technique of molecular-beam epitaxy to grow layers of CdTe on InSb and InSb on CdTe and have performed a detailed analysis of the layers and their interfaces using Auger depth profiling and reflection high-energy electron diffraction. We show that significant improvements in interfacial quality can be obtained by the proper choice of fluxes during growth. The use of a Cd/Te flux ratio of 3:1 ($J_{\text{Cd}}/J_{\text{Te}} = 3$) during the growth of CdTe has enabled epitaxy at a substrate temperature of 300 °C. Interfaces formed with this flux ratio are abrupt, in sharp contrast to those formed under stoichiometric flux conditions ($J_{\text{Cd}}/J_{\text{Te}} = 1$). Subsequent growth of InSb at a substrate temperature of 300 °C on thin CdTe epilayers (400 and 800 Å) is examined as a function of the InSb growth rate and Sb/In flux ratio. Quality of the interfaces shows a progressive improvement with increasing InSb growth rate.

I. INTRODUCTION

There is at present considerable interest in the InSb/CdTe material system. The mixed III-V/II-VI heterostructure has a near perfect lattice match ($\Delta a/a < 0.05\%$), which, combined with the large difference in band gaps between CdTe ($E_g = 1.44$ eV) and InSb ($E_g = 0.18$ eV), offers great potential for the fabrication of quantum-well lasers and detectors spanning the photon energy range 0.2–0.5 eV (2.5–6 μm). The small effective mass of electrons in InSb promises low-temperature HEMT structures with exceptionally high electron mobilities¹ and should allow quantization effects to be seen in significantly wider wells than with the more familiar material systems.

Attempts at experimental realization of multilayer structures have met with only limited success. Although good structural properties have been reported for CdTe layers grown by molecular-beam epitaxy (MBE) on InSb substrates^{2,3} and homoepitaxial layers⁴ and for InSb layers grown by MBE on (100) oriented CdTe substrates,⁵ recent studies^{6,11} of the growth of CdTe on InSb under typical MBE growth conditions have shown that the interfaces are complex due to a chemical reaction between InSb and CdTe. A study of the growth of InSb/CdTe multilayers has been reported,⁷ where reflection high-energy electron diffraction (RHEED) patterns indicated that individual layers grown at a substrate temperature of 220–240 °C were ordered. Although structurally good InSb epilayers have been reported⁸ for substrate temperatures in the range 225–275 °C, electrically active layers have only been reported⁹ for substrate temperatures in excess of 270 °C. To be useful for electronic device applications which involve tunneling, the thickness of individual layers of a multilayer device must be limited to several hundred angstroms. To facilitate growth, a substrate temperature must be found which results in high structural and electronic quality of both CdTe and InSb.

In this paper, we present results of a study of the MBE

growth of CdTe on InSb homoepitaxial layers, and of InSb on CdTe epilayers for potential applications in two-dimensional electron gas (2DEG) and electron tunneling devices. Auger depth profiling has been used to examine the layers and their interfaces. We show that significant improvements in interfacial quality can be obtained by the proper choice of fluxes during growth. CdTe layers have been grown on InSb for substrate temperatures of 200 and 300 °C under stoichiometric ($J_{\text{Cd}}/J_{\text{Te}} = 1$) and Cd-enhanced ($J_{\text{Cd}}/J_{\text{Te}} = 3$) flux conditions. The use of an enhanced Cd flux has enabled epitaxy of CdTe on InSb at 300 °C, a temperature which is compatible with the subsequent growth of electrically active InSb. Growth of InSb epilayers on thin layers (400 and 800 Å) of CdTe at a substrate temperature of 300 °C is examined as a function of InSb growth rate and Sb/In flux ratio. Interfaces are intermixed, but show a progressive improvement with increasing InSb growth rates.

II. EXPERIMENT

CdTe and InSb epitaxial layers were grown in a Varian 360 MBE system equipped with a quadrupole mass analyzer and *in situ* RHEED and flux monitoring facilities. Base pressure during growth was below 5×10^{-10} Torr. Prior to loading, InSb (100) substrates were solvent cleaned and mounted onto molybdenum support blocks using a colloidal suspension of graphite in alcohol. Immediately before growth the native oxide was removed from substrate surfaces by heating at 410 °C in an Sb₄ flux. A single effusion cell containing high-purity CdTe was used to provide a stoichiometric beam of Cd and Te₂ (Ref. 2) and was supplemented by a cell containing Cd for Cd-enhanced ($J_{\text{Cd}}/J_{\text{Te}} > 1$) studies. A relative measure of the flux from a given cell was obtained by interposing an ion gauge flux monitor into the molecular beam and relating the measured beam equivalent pressure to the cell temperature, molecular weight, and ionization efficiency of the beam species. Normal settings for the CdTe cell gave a total flux of 2.4×10^{14} atoms/cm²/s which corresponded to a homoepitaxial growth rate at $T_s = 220$ °C

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of approximately 1.7 \AA/s ($0.6 \mu\text{m/h}$). The Cd cell was set to supply its maximum safe flux of $2.6 \times 10^{14} \text{ atoms/cm}^2/\text{s}$ giving a flux ratio value $J_{\text{Cd}}/J_{\text{Te}} = 3$ during the Cd-enhanced growth experiments. Separate effusion cells containing high-purity In and Sb were used for growth of InSb. All InSb homoepitaxial and heteroepitaxial layers were grown at $T_s = 300^\circ\text{C}$ with growth rates in the range $0.3\text{--}2.0 \text{ \AA/s}$ ($0.1\text{--}0.7 \mu\text{m/h}$). Practical considerations related to the In cell temperature prevented growth of InSb with rates in excess of $0.7 \mu\text{m/h}$. $J_{\text{Sb}}/J_{\text{In}}$ was kept in the range $1.1\text{--}3.5$ to keep within the (2×2) Sb-stabilized surface reconstruction⁹ as indicated by RHEED.

Immediately following growth, each sample was transferred in air to a Physical Electronics 560 scanning Auger microscope configured with a double pass cylindrical-mirror analyzer and a differentially pumped and gettered argon ion gun. Depth profiles of the constituent species, as well as of carbon and oxygen, were obtained by alternate sputtering and data acquisition periods of 30 and 80 s, respectively. No carbon or oxygen recontamination was seen within the layers during the profile. To reduce artificial broadening of the growth interface, the sputter beam of 4-kV argon ions was incident at 40° from the sample surface normal with the sputter rate reduced to $20\text{--}30 \text{ \AA/min}$ by expanding to a $6 \times 6 \text{ mm}$ raster. The growths and sputter depths were limited to a few thin compositional periods to reduce roughness due to preferential ion etching of (100) InSb as observed in electron micrographs. The electron beam was used in spot mode and incident on the surface at 40° from the surface normal. An electron energy of 3 kV was used with a moderate and constant current of $0.16 \mu\text{A}$. Auger peaks overlapping the MNN transitions were minimized by collecting high energy resolution, $\Delta E/E = 0.4\%$, pulse count data from selectively contracted energy "windows."

The Auger analysis was calibrated in the usual manner. The analyzer energy scale was referenced to the known energy, 2 keV, of electrons backscattered from a sample surface while at its focal point, and the beam current was measured from the sample stage while biased to $+130 \text{ V}$. The current density of the argon beam was $600 \mu\text{A/cm}^2$ with FWHM of $550 \mu\text{m}$ and the sputter rates were referenced to thin-film standards and to bulk (110) CdTe. These agree well with our known MBE growth rates. Finally, Auger signal strength from bulk (100) InSb and (111) CdTe were used to judge stoichiometric conditions within the MBE layers.

III. RESULTS

Auger depth profiles of 300-s growths of CdTe under stoichiometric beam conditions ($J_{\text{Cd}}/J_{\text{Te}} = 1$) on 1000- \AA InSb homoepitaxial layers at substrate temperatures $T_s = 200$ and 300°C are shown in Fig. 1. For growth at $T_s = 200^\circ\text{C}$, the (2×2) InSb RHEED pattern converted to a streaked (2×1) CdTe pattern immediately after CdTe growth was initiated. The epilayer is stoichiometric with a thickness of approximately 500 \AA which corresponds closely to that calculated from flux measurements. Width of the interface is estimated to be $120\text{--}180 \text{ \AA}$, a value which is near the Auger resolution limit. For growth at $T_s = 300^\circ\text{C}$, the

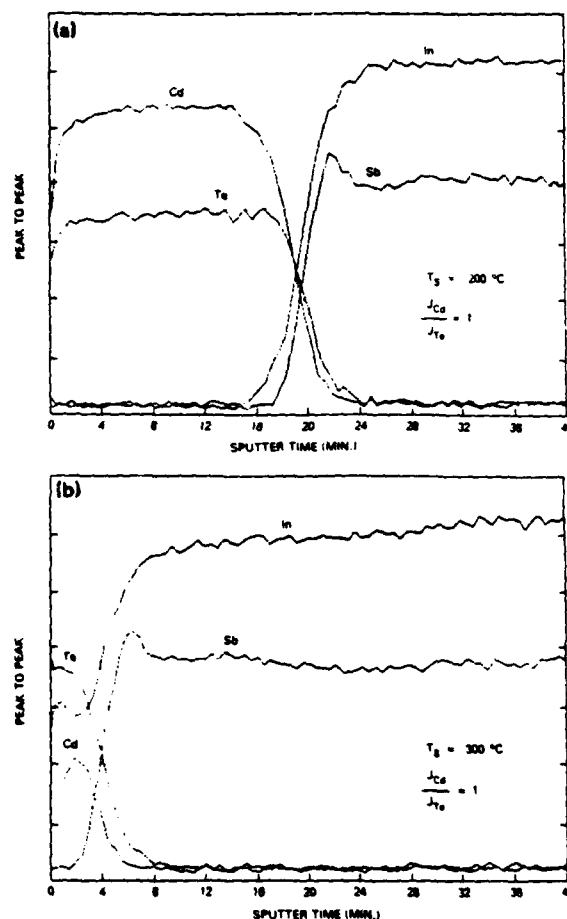


FIG. 1. Auger depth profiles of 300-s growths of CdTe on InSb homoepitaxial layers with $J_{\text{Cd}}/J_{\text{Te}} = 1$ for substrate temperatures of (a) 200°C and (b) 300°C .

(2×2) InSb RHEED pattern vanished immediately after CdTe growth was initiated, indicating a disordered growth. The resulting layer is deficient in Cd and rich in In and Te. Layer thickness is estimated to be 150 \AA . These results are similar to those recently reported by Mackey *et al.*,⁶ where indium telluride compounds were identified at the InSb/CdTe interface. It has been suggested that the lack of Cd incorporation in layers grown at 300°C may be attributed to a Cd deficiency on the growth surface, thereby allowing Te to react with the InSb. To overcome this Cd deficiency we grew layers of CdTe on InSb using an enhanced Cd flux. For $T_s = 300^\circ\text{C}$, the additional Cd flux has a dramatic effect on the quality of layers and interfaces. An Auger depth profile of a 300-s growth of CdTe using Cd-enhanced flux conditions ($J_{\text{Cd}}/J_{\text{Te}} = 3$) is shown in Fig. 2(a). The (2×2) InSb RHEED pattern converted to a streaked (2×1) pattern immediately after CdTe growth was initiated. The depth profiles show that the interfacial width for this sample is as abrupt as that grown under stoichiometric flux conditions at $T_s = 200^\circ\text{C}$. To establish that the high-quality epitaxy of thin CdTe layers grown at elevated temperatures is due to the enhanced Cd flux and not to the total Cd flux or growth rate, the temperature of the CdTe cell was set to give a Cd

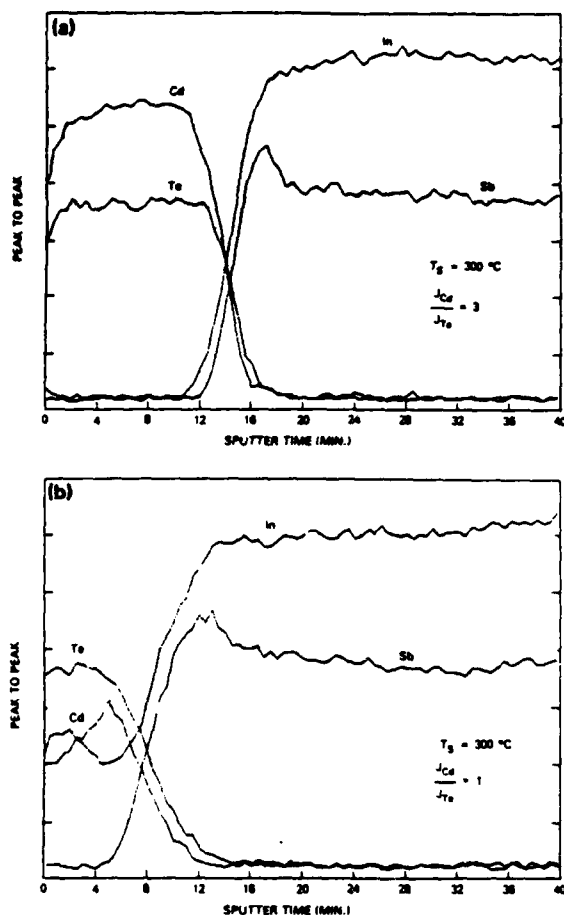


FIG. 2. Auger depth profiles of 300-s growths of CdTe on InSb homoepitaxial layers with substrate temperature of 300 °C for (a) $J_{Cd}/J_{Te} = 3$ and (b) $J_{Cd}/J_{Te} = 1$, with CdTe flux adjusted to give a Cd component equal to the total Cd flux in (a).

component equal to the total Cd flux used during the Cd-enhanced growth. An Auger depth profile of a 300-s growth of CdTe at $T_s = 300$ °C with $J_{Cd}/J_{Te} = 1$ and total flux of 7.2×10^{14} atoms/cm²/s (corresponding to a growth rate of 1.5 $\mu\text{m}/\text{h}$) is shown in Fig. 2(b). Again, the layer formed is rich in In and Te with severely degraded interfacial quality.

The ability to grow thin layers of CdTe on InSb at $T_s = 300$ °C made it possible to study the subsequent growth of InSb on these layers. Layer and interface quality of the films grown was examined as a function of the InSb growth rate and Sb/In flux ratio. Epitaxy of InSb on CdTe epilayers was not achieved for growth rates less than 0.15 $\mu\text{m}/\text{h}$ for J_{Sb}/J_{In} in the range 1.1–3.5. Immediately after initiation of growth of InSb, the streaked (2×1) CdTe RHEED pattern became spotty, an indication of three-dimensional nucleation. This spotted pattern remained unchanged throughout the growth period. Auger depth analysis revealed complete degradation of the 400-Å CdTe underlayer with severe intermixing throughout. For growth rates of 0.2 $\mu\text{m}/\text{h}$ or higher, the (2×1) CdTe RHEED pattern immediately became spotty, but gradually changed to the (2×2) Sb-stabilized pattern characteristic of homoepitaxial InSb. The time taken for the RHEED pattern to evolve was approximately 3, 2,

and 1 min for growth rates of 0.2, 0.4, and 0.7 $\mu\text{m}/\text{h}$, respectively. Auger analysis reveals that the degree of intermixing is independent of J_{Sb}/J_{In} but dependent on the InSb growth rate, with the highest growth rate resulting in the least intermixing. The decrease in In and Sb in the CdTe underlayer as the InSb growth rate is increased is evident in Fig. 3. For all growth rates, however, In and Sb are present throughout the 400-Å CdTe layer leading to a deterioration and broadening of the initial (CdTe on InSb) interface. To prevent this broadening of the initial interface, an 800-Å CdTe layer was grown. The depth profile of this structure, grown with our maximum InSb growth rate of 0.7 $\mu\text{m}/\text{h}$, is shown in Fig. 4. There is a distinct asymmetry between the first and second interfaces. Growth of the InSb epilayer has induced a Cd loss resulting in an In- and Te-rich interfacial region and incorporation of In and Sb throughout the CdTe layer. In a separate study, small shifts in energy and shape of Auger peaks

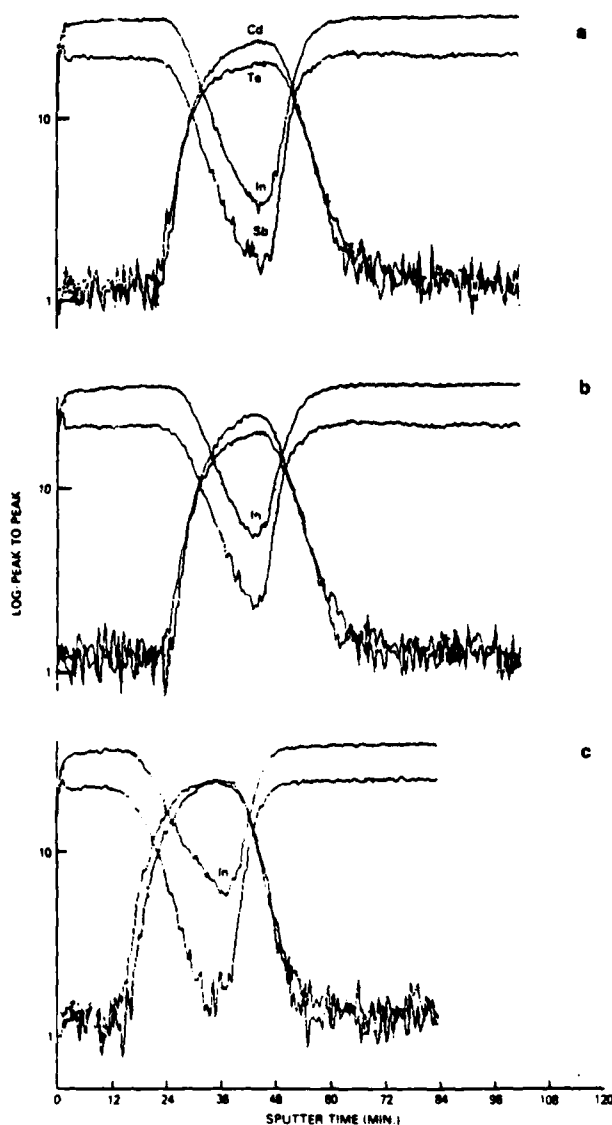


FIG. 3. Auger depth profile of an InSb epilayer grown with a substrate temperature of 300 °C on a 400-Å-thick CdTe layer with In and Sb fluxes corresponding to growth rates of (a) 0.7, (b) 0.4, and (c) 0.2 $\mu\text{m}/\text{h}$.

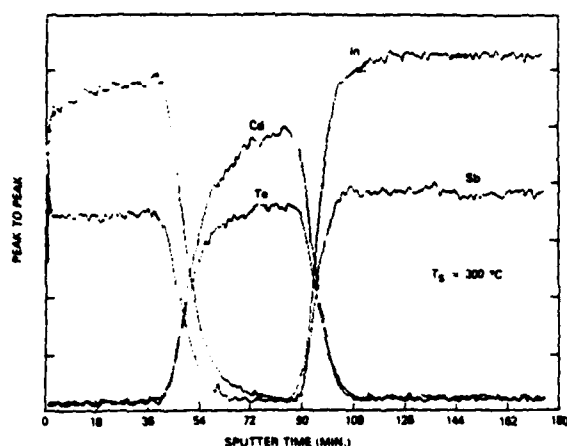
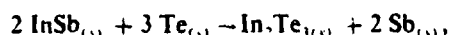


FIG. 4. Auger depth profile of an InSb/CdTe/InSb structure grown at a substrate temperature of 300 °C with an equivalent growth rate for InSb of 0.7 $\mu\text{m/h}$. The CdTe layer was grown to a thickness of ≈ 800 Å under Cd-enhanced flux conditions ($J_{\text{Cd}}/J_{\text{Te}} = 3$).

for the elements at each interface indicated similar electronic environments at each interface and the presence of excess antimony.

IV. DISCUSSION

We have shown that an attempt to grow CdTe on InSb at $T_s = 300$ °C under stoichiometric flux conditions results in a severely intermixed and disordered layer. The use of an enhanced Cd flux during growth has a dramatic effect, resulting in a CdTe layer which is epitaxial and an interface which is abrupt. This suggests that the residence time of Cd on the InSb surface is less than that of Te, resulting in a Cd deficiency at the growth surface and allowing Te to react with InSb to form indium telluride compounds, as suggested by



for which the change in enthalpy ΔH is -30.9 kcal. Use of an enhanced Cd flux clearly reduces or eliminates the Cd deficiency, ensuring that sufficient Cd is available on the InSb growth surface to bond with the incident Te and inhibit access of Te to the In. Thus, perhaps, the enhanced Cd flux need only be applied during the initial stages of growth to allow nucleation of the CdTe, after which the growth can be continued under stoichiometric beam conditions.

We believe that our data for growth at $T_s = 300$ °C of CdTe on InSb have implications for growth at 200 °C. Comparison of the interfacial width of layers grown on InSb at 200 °C with and without enhanced Cd flux indicates that an enhanced flux improves interfacial quality at this "typical" MBE growth temperature. We speculate that the formation of In_2Te_3 , seen⁶ in samples grown under similar conditions is reduced or eliminated by use of enhanced Cd flux during growth. The relatively low mobilities reported¹⁰ for a 2DEG at an InSb/CdTe interface may result from indium loss within the InSb due to indium telluride compound formation at the interface. Samples have been grown at $T_s = 200$ °C using an enhanced Cd flux and magnetotransport measurements

are in progress to assess the 2DEG transport characteristics. It would be of interest to extend our growth studies to include higher values of $J_{\text{Cd}}/J_{\text{Te}}$. The ability to grow CdTe layers on InSb at 200 °C with $J_{\text{Cd}}/J_{\text{Te}} = 3$ clearly suggests that higher flux ratios could be beneficial at higher growth temperatures.

For the growth of InSb on CdTe, the dependence of interfacial abruptness and epitaxy on the InSb growth rate suggests a competitive process between the intermixing and deterioration in the CdTe and the nucleation and growth of InSb. It is possible that broadening of the interface profile is caused by nonuniform sputtering of the InSb; however, the observed Cd deficiency indicates that deterioration of the CdTe has occurred during the InSb growth, most likely due to formation of indium telluride compounds. Results similar to those in Fig. 4 have also been obtained for a structure grown at 275 °C, indicating that atomically abrupt interfaces may not be obtainable for values of T_s compatible with growth of electrically active InSb.

V. CONCLUSION

In conclusion, we have found evidence of a strong chemical reaction which occurs at the CdTe/InSb interface during MBE growth at a substrate temperature of 300 °C. For growth of CdTe on InSb, we have presented a technique for suppressing this reaction by use of Cd-enhanced flux conditions. For growth of InSb on CdTe layers at a substrate temperature of 300 °C, we have found that epitaxy is strongly dependent on the InSb growth rate. Our results extend the substrate temperature range to $225 \leq T_s \leq 300$ °C, within which single-crystal layers of both CdTe and InSb can be grown at a fixed substrate temperature. We have shown that multilayer growth is possible at substrate temperatures compatible with electrically active InSb, but that thin period (200 Å) InSb/CdTe structures for tunneling devices may not be feasible due to breakdown of the CdTe barrier. However, the question of whether the chemical profiles presented are adequate for 2DEG or tunneling devices can be answered only after electrical transport measurements have been made.

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Molecular Beam Epitaxial Growth and Characterization of In_2Te_3

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Abstract

We report studies of the molecular beam epitaxial growth of In_2Te_3 . The unique structure of In_2Te_3 , with 1/3 of the In sublattice sites vacant, is of fundamental interest for molecular beam epitaxial growth dynamics. We show that thin film (500Å-7000Å) single crystal In_2Te_3 can be grown successfully on $\text{InSb}(100)$ homoepitaxial layers at substrate temperatures of 300 - 350°C and Te/In flux ratios of 3/2 - 5/2. Epitaxy has been monitored by reflection high energy electron diffraction and the stoichiometry of the grown layers assessed by Auger spectroscopy and energy dispersive X-ray analysis. Raman studies of the layers are presented and compared with a bulk In_2Te_3 standard. Crystal structure has been determined by X-ray diffraction using Weissenburg and oscillation photographs, confirming that the layers have a f.c.c. crystal structure with a lattice parameter of 18.50Å, in excellent agreement with the bulk value. Bandgap measurements have been performed on the layers by photoreflectance. We report a value for the α - In_2Te_3 bandgap of 1.19 eV and 1.31 eV at 300K and 77K respectively. Molecular beam epitaxial growth of InSb and CdTe on epitaxial In_2Te_3 films for fabrication of $\text{InSb}/\text{In}_2\text{Te}_3/\text{InSb}$ and $\text{InSb}/\text{In}_2\text{Te}_3/\text{CdTe}$ multilayers has been studied. Auger depth profiling of the resulting layers

shows severe intermixing into the In_2Te_3 . These results are supported by thermodynamic considerations of the $\text{InSb-In}_2\text{Te}_3$ interface.

Introduction

In the course of a study into the molecular beam epitaxial growth (MBE) of InSb/CdTe multilayers and superlattices, it has been found^{1,2} that there is a strong chemical reaction at the InSb/CdTe interface during growth at substrate temperatures compatible with the growth of high structural and electronic quality of both InSb and CdTe (275-300 °C).

Recent studies^{3,4,5,6} by soft X-ray photoemission and Raman spectroscopy of the heterojunction formed from the MBE growth of CdTe on InSb under stoichiometric flux conditions and growth temperatures of 200-300°C reveal that the InSb and CdTe react to form a complex interface identified to consist predominantly of In_2Te_3 and elemental Sb .

In_2Te_3 is a defect zinc blende type semiconductor with a bandgap of approximately 1 eV, lying between that of InSb ($E_g=0.18\text{eV}$) and CdTe ($E_g=1.44\text{eV}$). Thermodynamic considerations of the stability of In_2Te_3 with respect to InSb and CdTe ^{7,8} led us to examine the possibility of MBE growth of In_2Te_3 for use as a barrier between the CdTe and InSb to facilitate fabrication of InSb/CdTe multilayers, and the interesting possibility of exploiting the large difference in bandgaps between InSb and In_2Te_3 to grow $\text{InSb/In}_2\text{Te}_3$ multilayers for novel quantum well structures.

In this paper we present results of the study of the MBE growth of In_2Te_3 and characterization of the grown layers. We show that thin film single crystal In_2Te_3 can be grown on InSb (100) homoepitaxial layers. Reflection high-energy electron diffraction (RHEED) was used to monitor growth of the In_2Te_3 , and stoichiometry of the grown layers was assessed by Auger spectroscopy and energy dispersive X-ray analysis. Raman

spectroscopy of the In_2Te_3 MBE grown layer was compared with a In_2Te_3 bulk sample to verify chemical composition. The crystal structure of the In_2Te_3 layers was determined by X-ray diffraction techniques, confirming a f.c.c. structure with a lattice parameter of 18.50\AA , indicating that the MBE grown In_2Te_3 is in the alpha phase ($\alpha\text{-In}_2\text{Te}_3$)⁹ where the In sublattice vacancies are ordered. The bandgap of the In_2Te_3 layers has been determined by photoreflectance, yielding a value of 1.2eV at 300K and 1.31eV at 77K . As a preliminary investigation into $\text{InSb/In}_2\text{Te}_3/\text{CdTe}$ and $\text{InSb/In}_2\text{Te}_3$ multilayer structures, we have studied the MBE growth of InSb and CdTe on 500\AA thick In_2Te_3 epilayers. Auger depth profiling has been used to examine the layers and their interfaces. Results show a severe intermixing of both the CdTe and InSb into the In_2Te_3 layers. The experimental results are supported by thermodynamic considerations of the $\text{InSb-In}_2\text{Te}_3$ interface. Points of interest are discussed.

Experimental

The In_2Te_3 , InSb and CdTe layers were grown in a Varian 360 MBE system equipped with a quadrupole mass analyzer and *in situ* RHEED and flux monitoring facilities. Base pressure during growth was below 5×10^{-10} Torr. The $\text{InSb}(100)$ substrates were solvent cleaned and mounted onto molybdenum heating blocks using a colloidal suspension of graphite in alcohol. Immediately before growth the native oxide was removed from the substrate surfaces by heating at 410°C in an Sb_4 flux. A 1000\AA InSb buffer was grown on all substrates to ensure a consistent high quality $\text{InSb}(100)$ surface present for the growth of the Indium telluride.

Separate effusion cells containing high purity In , Sb , CdTe , Cd and Te were used for the growth of the InSb , CdTe and In_2Te_3 layers. A relative measure of the flux from a given cell was obtained by interposing an ion gauge flux monitor into the molecular beam and

relating the measured beam equivalent pressure to the cell temperature, molecular weight and ionization efficiency of the beam species.

Indium telluride growth was studied for substrate temperatures, T_s , within the range $300 \leq T_s \leq 350^\circ\text{C}$ and Te/In flux ratios ($J_{\text{Te}}/J_{\text{In}}$) between $1 < J_{\text{Te}}/J_{\text{In}} \leq 2$. The Te/In flux ratio range was restricted to help avoid the possibility of growth of the compounds InTe and In_2Te_5 . The In cell setting was kept constant throughout. Growth rates were estimated to be $0.2 \mu\text{m/hr}$ as calculated from the measured In flux, assuming a unity sticking coefficient for the In.

Following growth the samples were analyzed by Auger spectroscopy, and the atomic and weight percentages of the layers were assessed by energy dispersive X-ray analysis (EDX) using a standard facility with an accelerating voltage of 15 KV and a beam current of approximately 8×10^{-10} amps. All layers examined by EDX were $\sim 7000 \text{\AA}$ thick to ensure there would be no contributions from the InSb substrate appearing in the EDX spectra. A sample of bulk In_2Te_3 supplied by CERAC/PURE Inc was used for standardization purposes for both the Auger and EDX analysis. Selected layers were further characterized and studied by Raman spectroscopy, X-ray diffraction techniques and photoreflectance. During the course of our studies, it became apparent that the In_2Te_3 was oxidizing over a period of time when exposed to atmosphere. To avoid oxidation during transportation for the Raman studies, a 50\AA Sb cap layer was deposited at room temperature on the In_2Te_3 . It has been reported that Sb deposited under these conditions may be either amorphous or crystalline, dependant on the substrate and thickness¹⁰. RHEED studies during deposition of the Sb indicated that the Sb was amorphous. The Raman studies employed an Argon ion laser as an exciting source ($\lambda = 514.5 \text{ nm}$, $P = 40 \text{ mW}$) with the spectra taken in a back scattering geometry. A bulk In_2Te_3 sample⁴ grown by the horizontal Bridgeman technique at UC Cardiff was used as a standard for comparison. X-ray analysis of the layers used a single crystal diffractometer together with Weissenberg and oscillation photographs

utilizing Ca K α radiation. Photoreflectance measurements were used to measure the band-gap and were performed with a standard facility¹¹ that used a Ge or Si detector and a 6328Å He-Ne pump beam to produce the electric field modulation. Measurements were conducted at 300 and 77K in the vicinity of the direct gap, E_g .

InSb and CdTe were grown by MBE on thin film (500Å) In₂Te₃ epilayers at 300°C. The InSb and CdTe growth, together with the experimental details of the Auger depth profiling, has been described elsewhere¹.

Results

Initiating growth of the Indium telluride resulted in an immediate conversion from the Sb stabilized (2X2) InSb RHEED pattern¹² to a characteristic streaked pattern shown in Figure 1. For flux ratios $J_{Te}/J_{In} < 3/2$ this pattern became spotted and eventually vanished, indicative of a disordered growth, after approximately 300Å growth for all substrate temperatures studied. For growth with flux ratios $J_{Te}/J_{In} \geq 3/2$ and a substrate temperature of 300°C, the pattern remained streaked throughout the entire growth. Growth at substrate temperatures greater than 300°C required Te/In flux ratios greater than 3/2 to preserve the streaked two-dimensional growth pattern.

Energy dispersive X-ray spectra of samples grown at $T_s = 300^\circ\text{C}$ and flux ratios, $J_{Te}/J_{In} < 3/2$ and $J_{Te}/J_{In} \geq 3/2$ (Figure 2, (a) and (b) respectively) are compared with the spectrum obtained from a bulk In₂Te₃ reference standard. The spectra for layers grown at 300°C and flux ratios $J_{Te}/J_{In} \geq 3/2$ were identical to that obtained from the bulk In₂Te₃ standard, indicating that the stoichiometry of these layers was identical to that of the In₂Te₃ bulk standard. Layers grown with flux ratios $J_{Te}/J_{In} < 3/2$ were identified to be In rich.

Auger spectra of the MBE grown layers were compared with the Auger spectrum obtained for the In_2Te_3 bulk standard. The spectra obtained from samples grown at 300°C with flux ratios $J_{\text{Te}}/J_{\text{In}} \geq 3/2$ matched the spectrum obtained from the bulk standard. Samples grown with flux ratios $J_{\text{Te}}/J_{\text{In}} < 3/2$ were identified as In rich, correlating and supporting the results obtained from the EDX studies. An Auger spectrum from a stoichiometric In_2Te_3 layer is shown in Fig 3. High magnification SEM imaging of the samples showed a clear distinction between the stoichiometric and non-stoichiometric layers. The stoichiometric layers had a smooth surface morphology whilst non-stoichiometric layers were rough and structured with island-type precipitates. Further examination by EDX identified these precipitates as indium.

Raman studies of an 1100\AA thick MBE layer grown under $J_{\text{Te}}/J_{\text{In}} > 3/2$ and $T_s = 300^\circ\text{C}$ growth conditions were compared with the spectrum obtained from a bulk In_2Te_3 sample. Although the optical constants of In_2Te_3 are not well documented, the penetration depth of the laser light used in the studies was considered to be less than 1100\AA , ensuring that there would be no contributions from the InSb buffer and substrate appearing in the Raman spectrum. The spectra obtained for the MBE layer and In_2Te_3 bulk are shown in Fig. 4(a) and (b) respectively. Both spectra exhibit prominent peaks labeled A,B,C and D for the In_2Te_3 layer and A',B',C' and D' for the bulk material. The position of these features are in excellent agreement. Slight interference with A,B and C is caused by an additional background from the amorphous Sb, which has a characteristic broad band which peaks at 152 cm^{-1} ¹³. Additional features at 70 cm^{-1} can also be seen, although difficult to separate from the increasing background due to Rayleigh scattering from a slightly rough sample surface. Whilst little is known about the Raman spectrum of many of the indium tellurides, InTe has been reported¹⁴, and the spectrum differs substantially from Fig. 4 (a) and (b).

Further characterization of the MBE grown indium telluride and analysis of the crystal structure were obtained from X-ray diffraction, Weissenberg and oscillation photographs. Fig 5(a) and (b) show 2 θ diffractometer traces of a 1100Å thick In_2Te_3 layer grown on an $\text{InSb}(100)$ substrate for $\text{InSb}(200)$ and $\text{InSb}(400)$ reflections respectively. $\text{In}_2\text{Te}_3(600)$ and $\text{In}_2\text{Te}_3(12,00)$ reflections are observed in these traces suggesting that $\text{In}_2\text{Te}_3\langle 100 \rangle$ lies along $\text{InSb}\langle 100 \rangle$ direction with an out of plane lattice parameter of 18.50Å. This lattice parameter is in good agreement with a value of 18.486 reported⁹ for bulk In_2Te_3 . To confirm that the periodicity was 18.50Å in the plane of the epitaxial film, Weissenberg and oscillation photographs were taken. Figure 6 shows the oscillation photographs of $\text{In}_2\text{Te}_3/\text{InSb}(100)$ taken about $(01\bar{1})$ oscillation axis. First and second layers of InSb are observed with a repeat distance of 4.58Å ($6.48/\sqrt{2}$ Å). Weak and medium intensity layers are seen for In_2Te_3 . Zero to sixth layers are identifiable with a repeat distance of 13.08 Å ($18.50/\sqrt{2}$ Å), suggesting that the in plane lattice parameter of the film is 18.50Å. All the reflections can be indexed based on a f.c.c. lattice with lattice parameter of 18.50Å.

Photoreflectance spectra, measured in the vicinity of E_g are presented in Fig 7. The line shapes are qualitatively similar to those observed in other semiconductors¹¹ with a sharp oscillation near the transition energy; the broad feature below it is most likely due to absorption modulation of the light that is transmitted through the film and reflected from the $\text{In}_2\text{Te}_3/\text{InSb}$ interface¹⁵. The maximum in the spectrum is assumed to represent the transition energy and found to be 1.19 ± 0.02 eV and 1.31 ± 0.015 eV, at 300 and 77K respectively.

The ability to grow thin film In_2Te_3 made it possible to study the subsequent growth of InSb and CdTe on these layers. An Auger depth profile of a 500Å In_2Te_3 layer grown on a InSb homoepitaxial layer is shown in Fig. 8. The interface is seen to be abrupt and estimated to be less than 100Å, with no evidence of intermixing between the InSb and In_2Te_3 layers. A 800-s InSb growth was attempted on a 500Å In_2Te_3 layer with $T_s=300$

°C and a growth rate of 0.6 Å/s to give a nominal growth of 500 Å. The RHEED pattern showed no change from the characteristic In_2Te_3 pattern (Fig. 1) upon initiating, and during growth of the InSb . The Auger depth profile of the resulting layer is shown in Fig. 9(a). There is no evidence of InSb layer growth. Comparison with Fig. 8 indicates that the layer is In rich with respect to In_2Te_3 and the interface broader. A 300-s CdTe growth was attempted on a 500 Å In_2Te_3 layer at $T_s=300^\circ\text{C}$ under stoichiometric flux conditions ($J_{\text{Cd}}/J_{\text{Te}}=1$) and nominal growth rate of 1.85 Å/s. The RHEED pattern remained unchanged from the In_2Te_3 pattern during growth of the CdTe . The resulting Auger depth profile is shown in Figure 9(b). No CdTe layer growth is evident. However, Cd has been incorporated through approximately 400 Å of the indium telluride layer, suggesting the formation of an alloy. A similar CdTe growth was attempted, with a Cd/Te flux ratio $J_{\text{Cd}}/J_{\text{Te}}=3/1$. The RHEED pattern remained unchanged from the In_2Te_3 pattern during growth. The resulting profile is shown in Fig. 9(c). In this case the Cd has mixed uniformly throughout the indium telluride layer. Initial analyses of the Auger data using sensitivity factors suggest that the resulting layer has ratios Cd:In:Te of 1:2:4

Discussion

We have shown that single crystal In_2Te_3 can be grown epitaxially on $\text{InSb}(100)$ at substrate temperatures $300 \leq T_s \leq 350$ and flux ratios $3/2 \leq J_{\text{Te}}/J_{\text{In}} < 5/2$. For a given growth temperature, there is a minimum Te/In flux ratio required for epitaxial growth (3/2 at 300°C), below which the layers are non-epitaxial and indium rich. The minimum Te/In flux ratio increases as the growth temperature is increased, suggesting that the ratio in the Te/In sticking coefficients decrease with increasing growth temperature.

The Weissenberg and oscillation photographs indicate that the In_2Te_3 layers are in the α -phase, where the cation vacancies are distributed uniformly throughout the In sublattice. It

is of fundamental interest for MBE growth dynamics to ascertain whether the ordered vacancy structure is formed on the growth surface during epitaxy, or if the vacancy distribution on the growth surface is random and there is a subsequent rearrangement of the In sublattice within the layer. A detailed analysis of the RHEED pattern during growth may help to clarify these points.

We report a value for the bandgap of the α -phase of In_2Te_3 to be $1.19 \pm 0.02 \text{ eV}$ and $1.31 \pm 0.015 \text{ eV}$, at 300 and 77K, respectively as measured by photoreflectance. As far as we are aware, this is the first direct measurement of the band-gap of $\alpha\text{-In}_2\text{Te}_3$. A value of 1.12 eV has been deduced from electrical measurements¹⁶. Other 300K band gap values reported range from 1.01 eV for the β -phase, determined from absorption measurements¹⁷, to 1.2 eV determined from the reflectivity¹⁸; pseudopotential calculations yield a band gap of 1.3 eV for the β -phase¹⁹.

We have shown that an attempt to grow an epitaxial layer of either InSb or CdTe on In_2Te_3 layers at growth temperatures compatible with InSb/ In_2Te_3 and CdTe/ In_2Te_3 /InSb multilayer growth (300°C) results in severe intermixing in the In_2Te_3 layer with no InSb or CdTe layer growth. RHEED studies indicate that the layers formed are crystalline. For InSb growth on In_2Te_3 Auger depth studies show In incorporation into the In_2Te_3 layer. In contrast an abrupt interface is observed for the growth of In_2Te_3 on InSb. Consideration of the thermodynamics of the In_2Te_3 -InSb interface can help to explain the heterogeneity of the two growths. For this, we ignore the fact the reactants may be dimers or tetramers (i.e. Te_2 , Sb_4), assuming that the molecular beam species degenerate into the monomer upon contact with the growth surface. As such the deduced change in enthalpy should only be considered approximate. For the growth of In_2Te_3 on InSb we can consider the following reaction where In and Te are present on an InSb surface.



for which the change in enthalpy ΔH is -45.8 kcal^{20} . Our growth conditions were carefully controlled by tailoring $J_{\text{Te}}/J_{\text{In}}$ to ensure In_2Te_3 formation rather than InTe or In_2Te_5 , and we will not consider these other possible reactions here. For the growth of InSb on In_2Te_3 we consider In and Sb present on an In_2Te_3 surface.



where the In and Sb react to form InSb . However, if the Sb has a short residence lifetime with respect to the In , the growth surface may be Sb deficient and we should consider the following possible reactions.



For reactions (3) and (4) the elemental In converts In_2Te_3 to InTe or In_2Te respectively. The experimental results indicating the inability to grow InSb on In_2Te_3 , with the incorporation of In into the In_2Te_3 layer suggests that the MBE growth of InSb on In_2Te_3 may be described by a reaction such as (3) or (4).

Whilst these thermodynamic considerations are non-rigorous, they suggest that InSb growth may be possible on In_2Te_3 if sufficient Sb is present on the In_2Te_3 growth surface to bond with the available In . Once InSb growth has been initiated on the In_2Te_3 , the resulting interface should be stable, since no chemical equation with a negative change in enthalpy can be written with InSb and In_2Te_3 as the reactants. This thermodynamic consideration is supported by the abrupt interface observed for the growth of In_2Te_3 on InSb (Figure 8).

For CdTe growth on In_2Te_3 there is severe Cd interdiffusion. Increasing the Cd flux results in a uniform incorporation of the Cd throughout the indium telluride layer. Preliminary Auger data suggests that the layer so formed may be a ternary alloy such as CdIn_2Te_4 . Further analysis by Raman and X-ray will help to clarify these points. The

readiness of Cd to interdiffuse into the In_2Te_3 layer supports the notion that the interfacial layer formed during growth of CdTe on InSb under conventional MBE growth conditions is complex and is unlikely to consist of merely a thin layer of In_2Te_3 and elemental Sb.

While these studies suggest that the successful growth of InSb/ In_2Te_3 and CdTe/ In_2Te_3 multilayers is unlikely, the results shed additional insight into the MBE growth of mixed systems. Recent studies^{1,8} of the growth of CdTe on InSb have shown that simple thermodynamic considerations⁸ are applicable to the MBE growth of this II-VI/III-V mixed system. The results presented in this paper also suggest that simple thermodynamic concepts can be successfully applied to the MBE growth of a mixed III-V/III-VI system. We believe that these thermodynamic concepts combined with considerations of the surface residence lifetimes of the incident beam species will prove to be of fundamental importance in predicting and optimizing growth conditions for the MBE of other mixed multilayer systems.

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20 Change of enthalpy calculated from heats of formation obtained from Ref.7

Figure captions

Figure 1

Reflection high energy electron diffraction pattern observed along $[01\bar{1}]$ azimuth during growth of In_2Te_3 with $T_s=300^\circ\text{C}$ and $J_{\text{Te}}/J_{\text{In}} = 3/2$.

Figure 2

Energy dispersive X-ray spectra of 7000Å thick indium telluride layers grown by MBE at a growth temperature of 300°C and with flux ratios (a) $J_{\text{Te}}/J_{\text{In}} < 3/2$ and (b) $J_{\text{Te}}/J_{\text{In}} \geq 3/2$. The spectra are compared with the spectrum obtained from a In_2Te_3 bulk standard.

Figure 3

Auger spectrum of MBE grown In_2Te_3 layer.

Figure 4

Raman spectra ($\lambda=514.5\text{nm}$, $T=80\text{K}$) of (a) 1100Å thick In_2Te_3 layer capped with 50Å of Sb. (b) In_2Te_3 bulk standard.

Figure 5

Diffraction traces of 1100Å thick In_2Te_3 layer grown on $\text{InSb}(100)$ substrate for (a) $\text{InSb}(200)$ reflection. (b) $\text{InSb}(400)$ reflection.

Figure 6

Oscillation pattern of $\text{In}_2\text{Te}_3/\text{InSb}(100)$ taken about $(01\bar{1})$ oscillation axis. All reflections can be indexed based on a f.c.c. lattice with lattice parameter of 18.50Å.

Figure 7

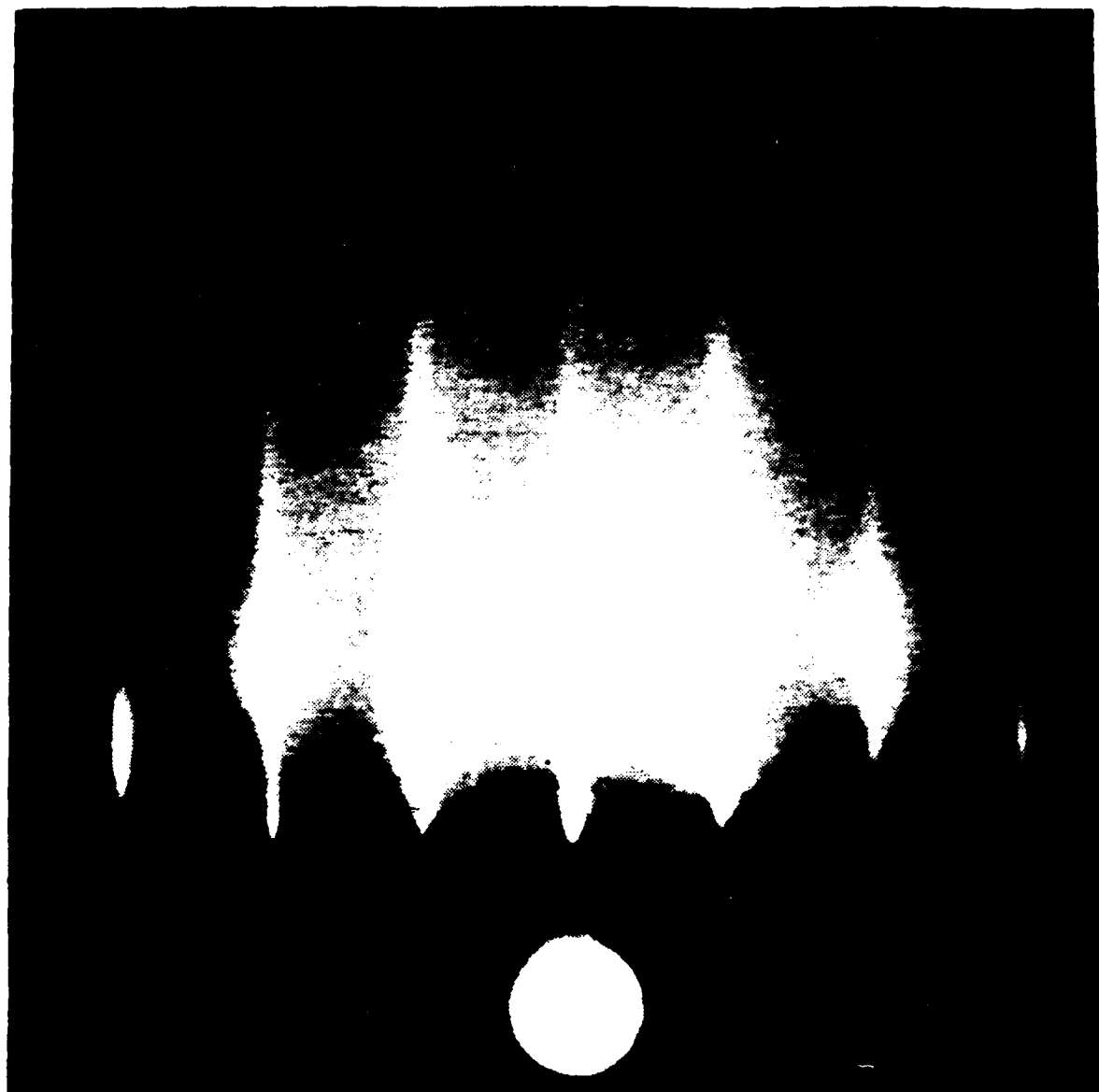
Photoreflectance spectra of 1100Å In_2Te_3 layer. The transition energy is $1.19 \pm 0.02\text{eV}$ and $1.31 \pm 0.015\text{eV}$, at 300K and 77K respectively.

Figure 8

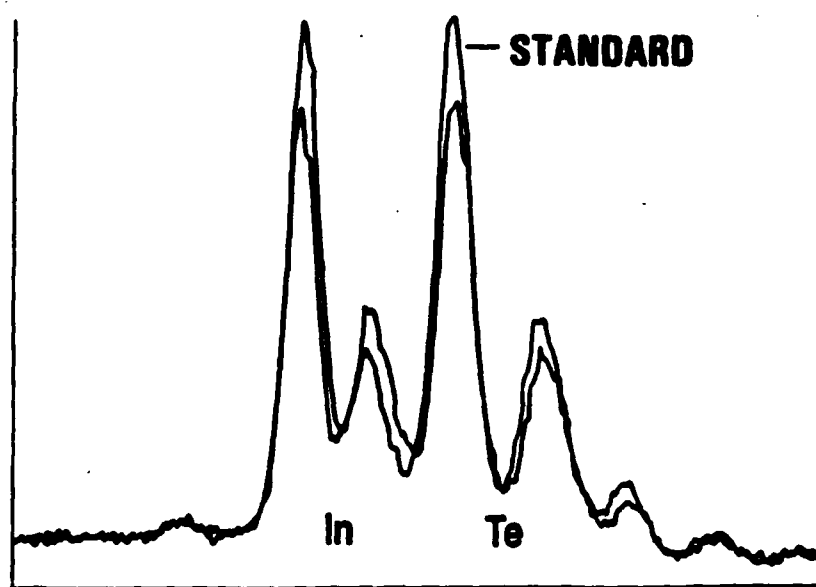
Auger depth profile of 500Å In_2Te_3 layer grown on an InSb homoepitaxial layer.

Figure 9

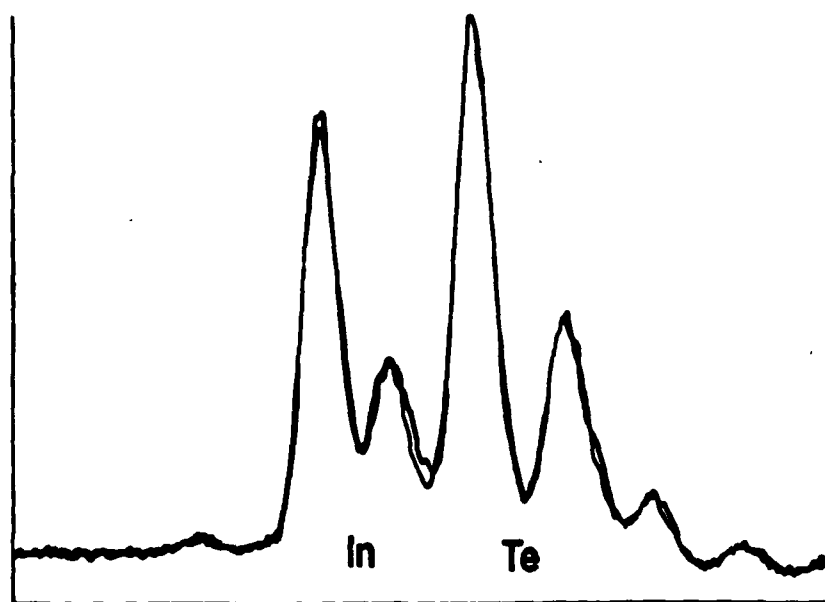
Auger depth profiles of the attempted growth at 300°C of (a) InSb , (b) CdTe with $J_{\text{Cd}}/J_{\text{Te}} = 1$, (c) CdTe with $J_{\text{Cd}}/J_{\text{Te}} = 3$, on 500Å thick In_2Te_3 layers. The estimated nominal thickness of the InSb and CdTe growths was 500Å.



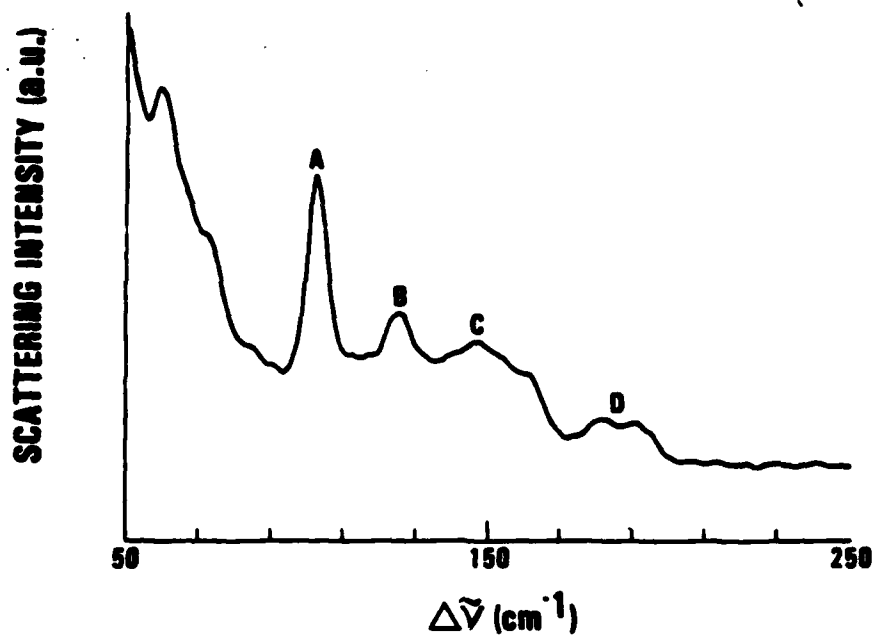
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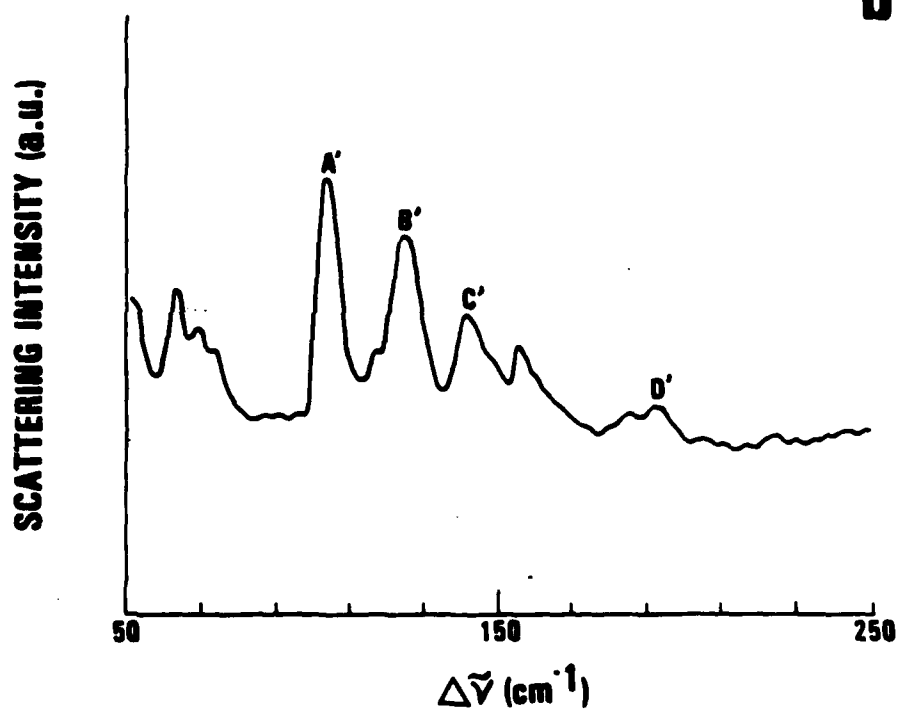
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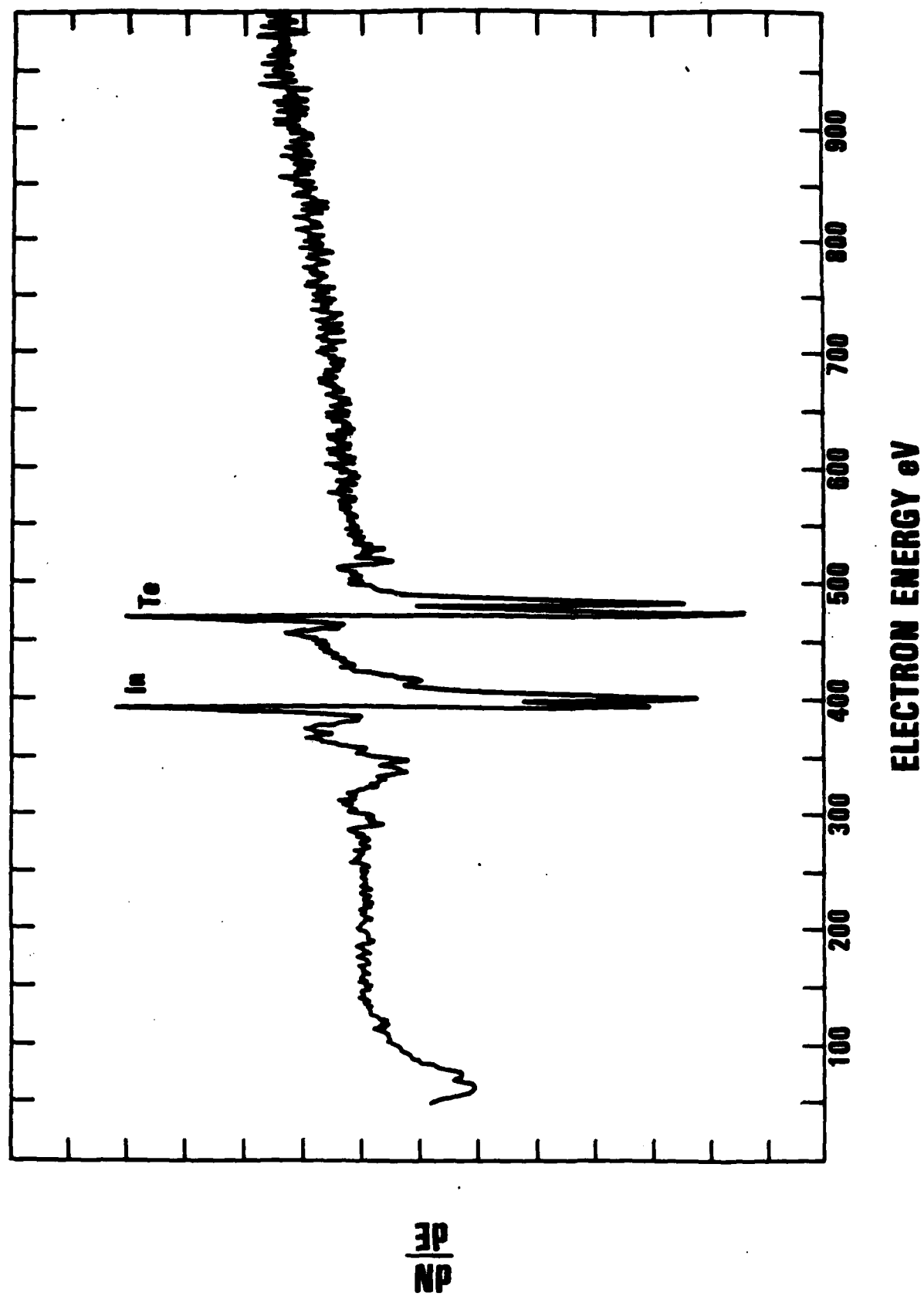


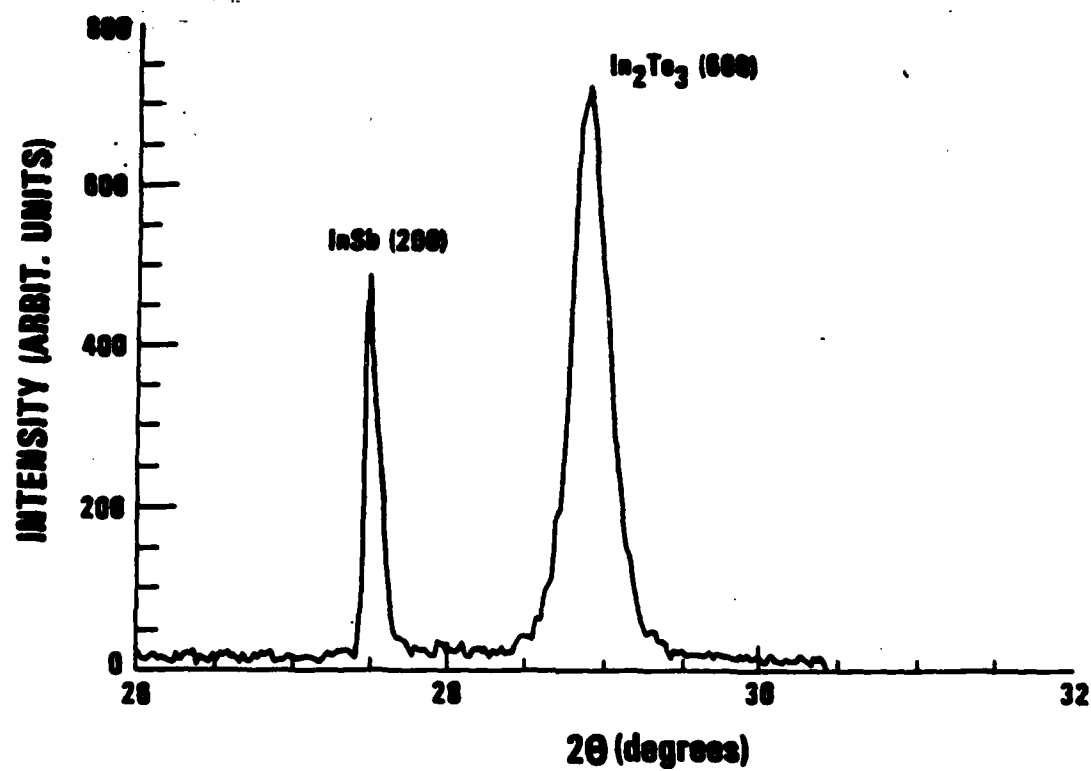
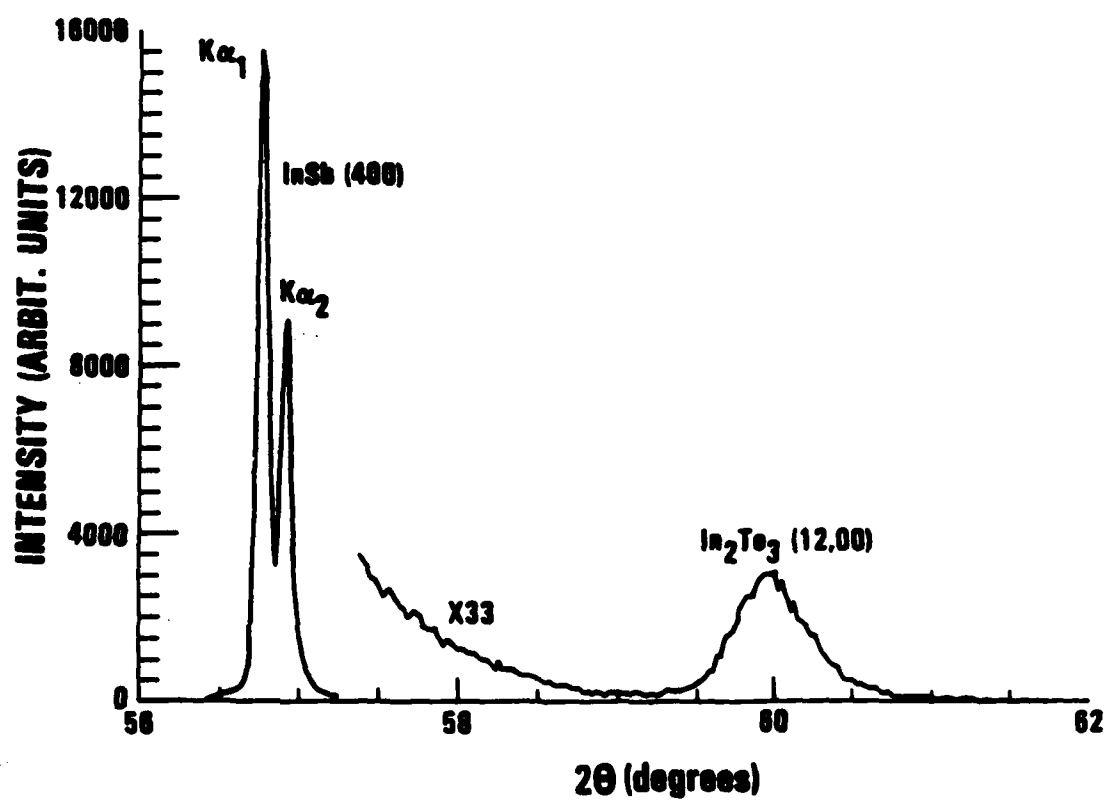
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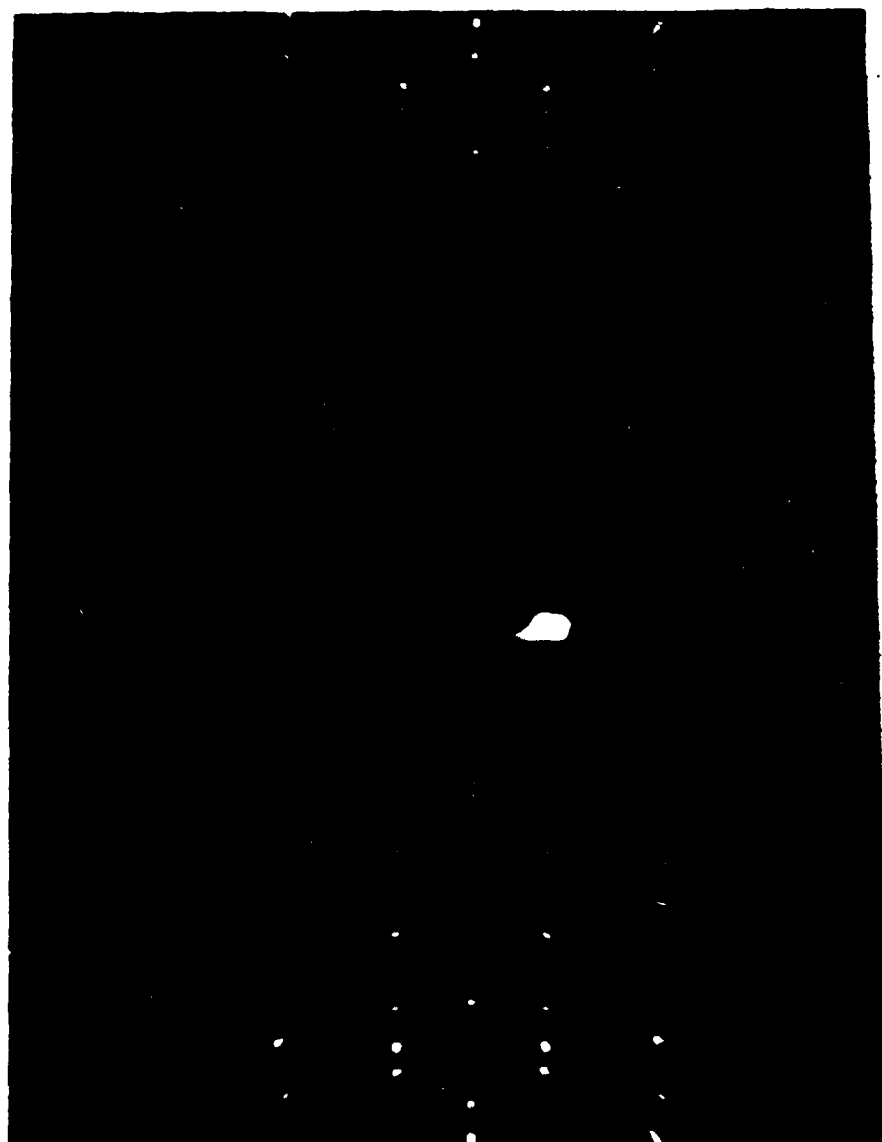


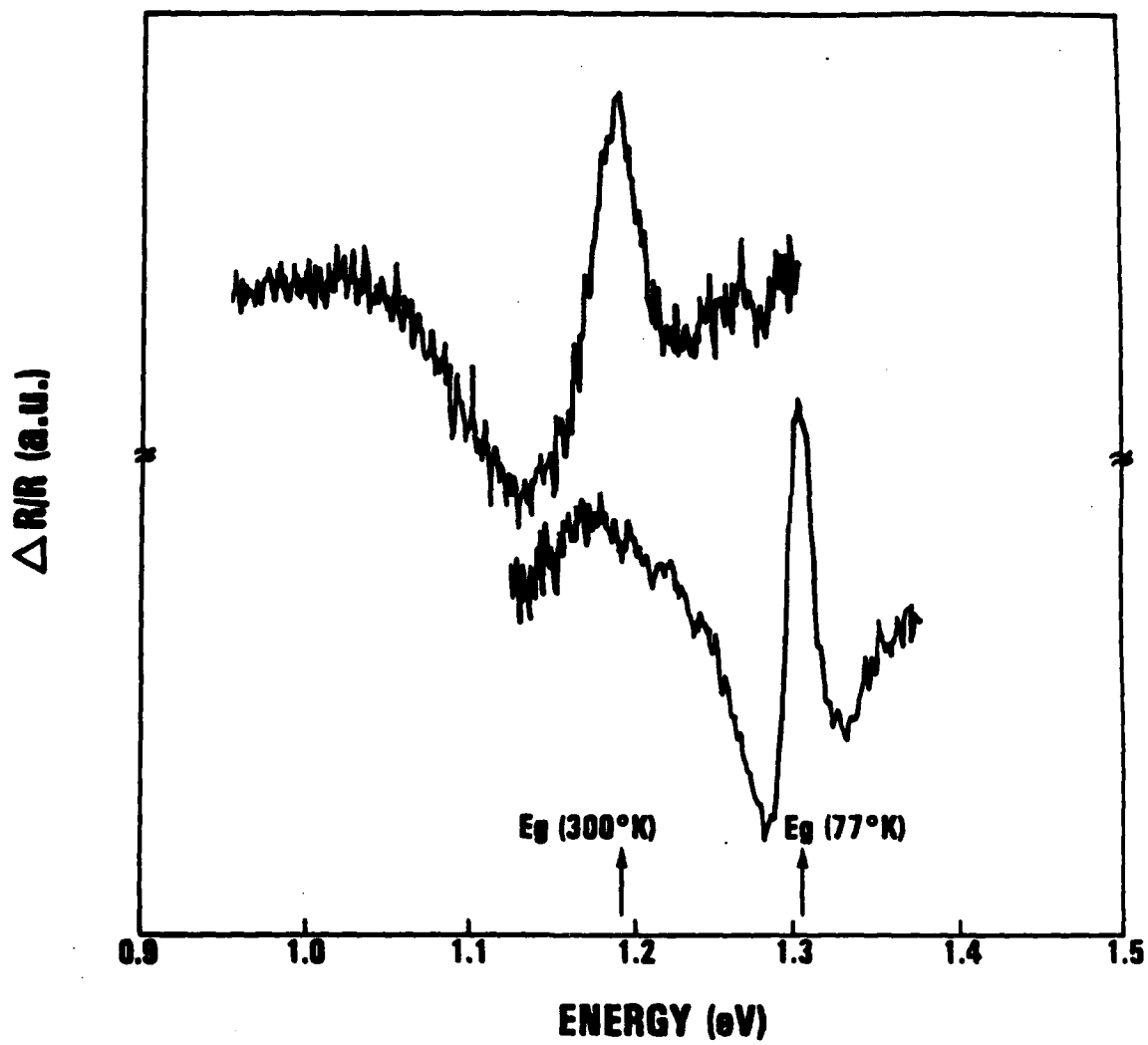
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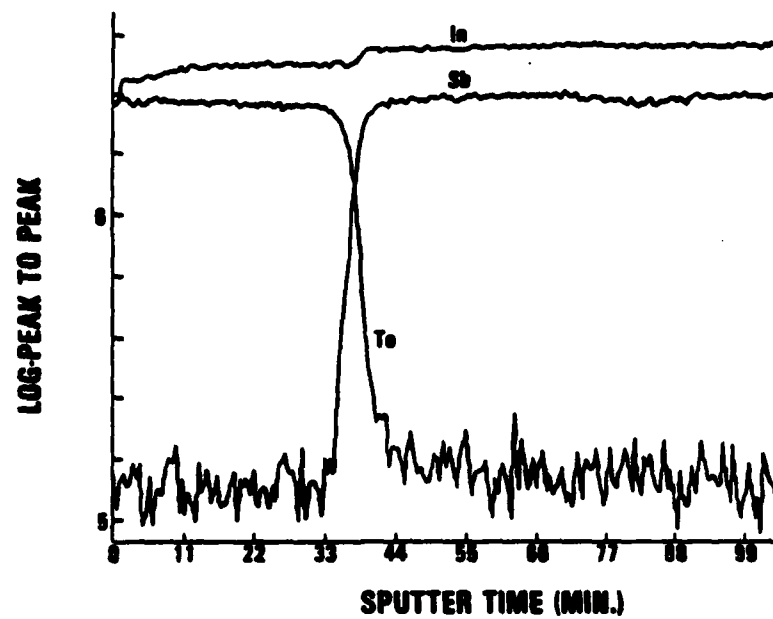


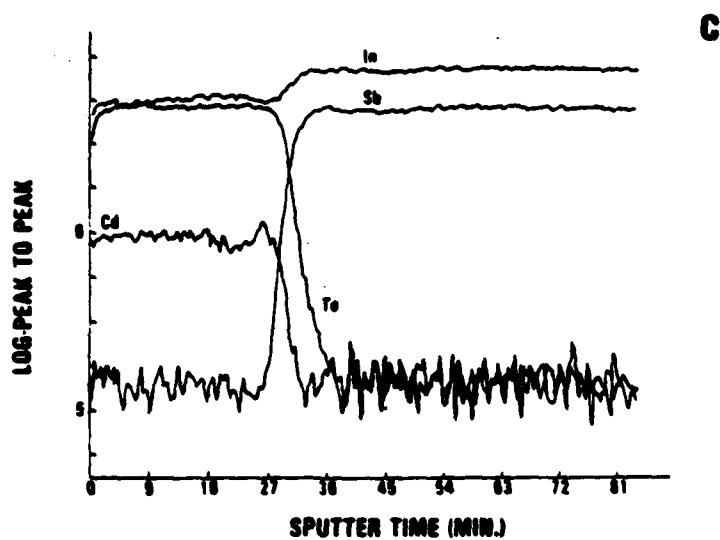
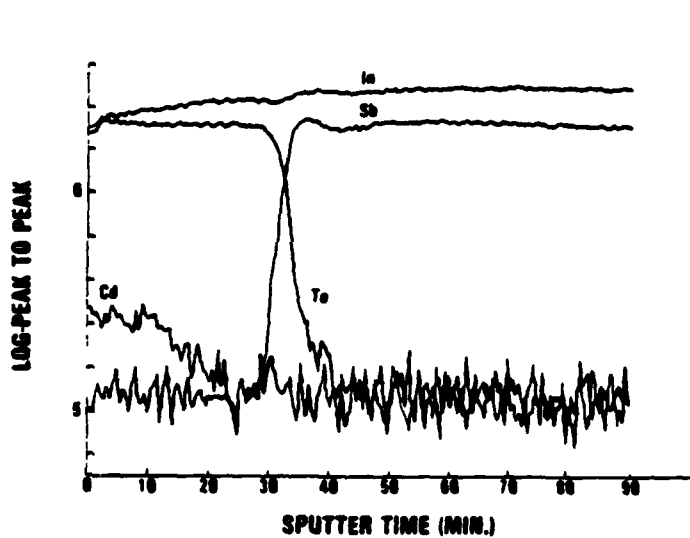
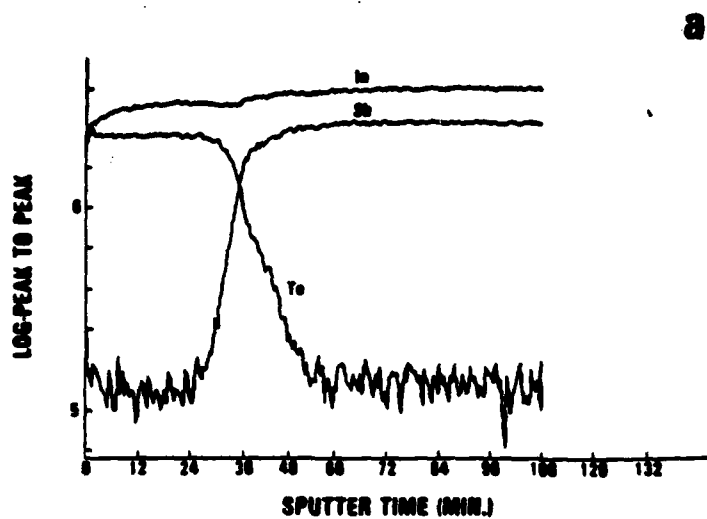


a**b**









Molecular Beam Epitaxial Growth and X-ray Characterization of (Zn,Cd)Te/CdTe Strained Layer Superlattices

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ABSTRACT

The need for a suitable low defect density, lattice matched substrate or substrate/buffer combination for the molecular beam epitaxial growth of thin film (Hg,Cd)Te has led us to examine the use of (Zn,Cd)Te/CdTe strained layer superlattices. (Zn,Cd)Te/CdTe strained layer superlattices have been grown by molecular beam epitaxy on CdTe(100), InSb(100) and GaAs(100) substrates and substrate/buffer combinations with period thicknesses ranging from 90Å to 330Å and with total superlattice thicknesses of 0.8µm and 1.6µm. The ratio in thickness between the (Zn,Cd)Te and CdTe strained layers is tailored to allow the in plane lattice parameter of the free standing superlattice to match that of Hg_{0.8}Cd_{0.2}Te. The resulting structures have been characterized by X-ray diffraction techniques. Multiple satellite peaks are observed indicating that these superlattices can be grown with a high structural quality. The structural quality is found to be independent of the superlattice period but dependent on the originating substrate.

INTRODUCTION

There is at present, a need to produce large area, high quality (Hg,Cd)Te for the fabrication of infrared detectors for the 8-12 μ m region. In view of the high defect content of bulk grown (Hg,Cd)Te, there is currently a great deal of interest and effort directed to the production of this alloy in thin film form by Molecular Beam Epitaxy (MBE).

One of the problems associated with the MBE of (Hg,Cd)Te is the current lack of a suitable substrate material. Whilst CdTe has the advantage of a close lattice match to (Hg,Cd)Te as well as similar metallurgical properties, it suffers from an unacceptably large defect concentration¹. Because of this alternative materials such as GaAs or InSb are also employed as substrates. However, the lattice mismatch between (Hg,Cd)Te and these materials necessitates the growth of a thick (Zn,Cd)Te buffer layer to provide a lattice matched template.

This need for a low defect density, lattice matched material has led us to examine the use of (Zn,Cd)Te/CdTe strained layer superlattices as a possible improvement to single (Zn,Cd)Te epilayer growth, by reducing the density of dislocations by bending and blocking threading dislocations propagated from the substrate and substrate/buffer interface. Strained layer superlattices incorporated in buffer layers have been shown to greatly reduce the number of dislocations threading from a substrate to an overlayer in III-V systems². This concept has yet to be demonstrated in II-VI systems. Whilst CdTe/ZnTe strained layer superlattices have been grown and reported^{3,4,5,6} it is not possible, as will be discussed later, to tailor the lattice parameter of the free standing superlattice to match that of (Hg,Cd)Te, and as such one must employ the ternary/binary (Zn,Cd)Te/CdTe strained layer superlattice structure.

To avoid the generation of misfit dislocations in the (Hg,Cd)Te epilayer we require that the lattice parameter of the free standing superlattice match that of $\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}$ which is exactly lattice matched to $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$, whose composition is tuned to include the atmospheric window at $12.4\mu\text{m}$. In addition the individual layers of the superlattice must be kept below a critical thickness^{7,8,9}, h_c , to ensure that the superlattice is coherently strained. This critical thickness is a function of the misfit, f , between the lattice parameters of the individual layers. For the $\text{Zn}_x\text{Cd}_{1-x}\text{Te}/\text{CdTe}$ system $f = -0.381x/a_{\text{CdTe}}$, where a_{CdTe} is the lattice parameter of bulk CdTe. For x values ranging from 0 to 1, f will vary from 0 to 6%. In order to maintain an in-plane lattice match with $\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}$, the ratio in the $\text{Zn}_x\text{Cd}_{1-x}\text{Te}:\text{CdTe}$ layer thicknesses must be tailored according to the following relation

$$1 + G_{\text{CdTe}}L_{\text{CdTe}} / (G_{\text{CdTe}}L_{\text{CdTe}} + G_{\text{ZnCdTe}}L_{\text{ZnCdTe}}) = a_{\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}} / a_{\text{CdTe}}$$

where $a_{\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}}$ is the bulk lattice parameter of $\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}$, G the shear moduli, and L the thickness of the respective $\text{Zn}_x\text{Cd}_{1-x}\text{Te}$ and CdTe layers.

From the above relation we find that if the superlattice were to consist of a ZnTe/CdTe binary system the ZnTe:CdTe layer thickness ratio would have to be 1:42, for the in-plane lattice parameter to match that of $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$. Assuming that ZnTe layers can be grown as thin as 10\AA , the CdTe layers must be greater than 400\AA , far in excess of the critical thickness for this system¹⁰. Thus strained layer superlattices matched to $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$ must consist of a (Zn,Cd)Te/CdTe structure. Whilst the growth of a ternary introduces complications in growth, a distinct advantage of a ternary/binary over a binary/binary system is that the misfit, and thus the strain, can be varied between the composite materials of the superlattice while still maintaining the required free standing lattice parameter by tailoring the layer thickness ratio. In

addition, layers grown with less misfit allow greater flexibility in the choice of layer thickness, since the critical thickness h_c increases as the misfit decreases.

An estimate for the critical thickness in these structures was obtained through consideration of a number of theoretical models. (Van der Merwe^{7,10}, Matthews^{8,10} and Bean^{9,10}). We find that the smallest critical thickness as a function of misfit is predicted from the Van der Merwe energy balancing model (for misfits below approximately 3.5%). In this study superlattices were grown with equal layer thicknesses of $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ and $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$. Additional $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ superlattices were grown with layer thickness ratios of 1:3, to allow the lattice parameter of the free standing superlattice to match that of $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$. For our maximum misfit superlattice ($\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$) the Van der Merwe model predicts a critical thickness of approximately 250Å. Thus, the maximum period thickness for the $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ superlattices is therefore approx 500Å and 330Å for the 1:1 and 1:3 structures respectively. For consistency and comparative purposes no samples were grown with period thicknesses > 330Å.

In this paper we present the initial results of the study of the MBE growth of $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ and $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ strained layer superlattices on $\text{InSb}(100)$, $\text{CdTe}(100)$, and $\text{GaAs}(100)$ substrates. A series of superlattices has been grown with period thicknesses ranging from 85Å to 330Å and with total superlattice thicknesses of 0.8μm and 1.6μm. The resulting structures have been characterized by X-ray diffraction techniques. Superlattice quality is compared for the various substrates, Zn compositions and layer thicknesses. Points of interest are discussed.

EXPERIMENTAL

Superlattice samples were grown in a Varian 360 MBE system equipped with quadrupole mass analyses and *in situ* reflection high energy diffraction (RHEED) and flux monitoring facilities. Base pressure during growth was below 5×10^{-10} torr. A single effusion cell containing high purity CdTe was used to provide a stoichiometric beam of Cd and Te_2 ¹¹ for the growth of CdTe and was supplemented by an effusion cell containing Zn for the growth of (Cd,Zn)Te. All epilayers and superlattices were grown at substrate temperatures of 270°C. Calibration of the Zn cell setting against Zn content for the growth of the (Cd,Zn)Te layers was determined by growing a series of (Cd,Zn)Te layers with various Zn cell settings and determining their Zn content by energy dispersive X-ray spectroscopy and X-ray diffraction analysis.

All substrates were solvent cleaned, etched and mounted onto molybdenum support blocks. The CdTe(100) substrates were heat treated at 310°C to remove the native oxide. A 1000Å CdTe epilayer was grown to ensure a consistent quality layer present for the growth of the superlattices. The InSb(100) substrates were heated at 410°C in an Sb_4 flux to remove the native oxide. Following this a 1000Å InSb buffer was grown to ensure a consistent high quality InSb(100) starting surface. Details of the InSb growth have been described elsewhere¹². The GaAs(100) substrates were heated to 610°C and held at this temperature until the native oxide had been desorbed, as indicated by RHEED. To ensure the growth of a (100) oriented CdTe epilayer, the technique described by Kolodziejski et al¹³ was employed where the CdTe cell was opened whilst the substrate was at the elevated temperature of 610°C. The substrate temperature was then dropped to the desired growth temperature of 270°C and a 1000Å CdTe epilayer was grown prior to growth of the superlattice. Confirmation of (100) oriented epitaxy was obtained by RHEED.

Following growth the samples were analyzed by X-ray diffraction using a standard single crystal diffractometer.

RESULTS

The quality of the multilayers as a function of the period thickness was assessed by growing a series of $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ superlattices on $\text{InSb}(100)$ substrates with period thicknesses of 85Å, 165Å, 210Å and 330Å. The $(\text{Zn,Cd})\text{Te}:\text{CdTe}$ layer thickness ratio was 1:1 for these samples with the total number of periods in each sample adjusted to maintain a constant superlattice thickness of 1.6µm. Fig. 1 shows the diffraction spectra from three of the superlattices, with nominal period thicknesses of 85Å, 165Å and 220Å. (A similar set of superlattices was grown, with a total superlattice thickness of 0.8µm. No noticeable difference in the diffraction spectra was observed for the two thicknesses). The $n=+1$ and $n=-1$ satellite peaks are clearly resolved and show a systematic increase in the peak separation with decrease in period thickness. From the separation of the peaks we calculate the period thicknesses to be 87Å, 165Å and 210Å, in excellent agreement with the values estimated from growth rates. The $\text{Cu K}\alpha_1$ and $\text{K}\alpha_2$ lines are clearly resolved, indicating a high structural quality. The decrease in the satellite intensity as the period thickness is decreased is due to an envelope modulation¹⁴ of the peaks; this modulation is also responsible for the extinction of satellite peaks $> /1/$ in these structures. The central peak of the satellites ($n=0$) is found to lie at a value corresponding with an average out-of-plane lattice parameter for the superlattice equal to that of $\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}$, in good agreement with the intended growth structure. The spectrum obtained from the 330Å thick period sample is shown in Fig. 2(b) and is compared with the spectrum obtained from an identical structure grown with $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ layers. The $n=+2$ and $n=-2$ satellite

peaks can be observed in the 330Å period samples due to the decrease in the satellite separation for this thicker period, moving the peaks within the modulating envelope. Both superlattice spectra indicate a high quality structure as is evident from the resolution of the Cu $K\alpha_1$ and $K\alpha_2$ lines. Comparing the relative positions of the satellite peaks in the two samples, one can observe the general shift toward higher angles for the $Zn_{0.16}Cd_{0.84}Te/CdTe$ sample. The central peaks ($n=0$) lie at values corresponding with an average out of plane lattice parameter equal to that of $Zn_{0.04}Cd_{0.96}Te$ and $Zn_{0.08}Cd_{0.92}Te$ for the $Zn_{0.08}Cd_{0.92}Te/CdTe$ and $Zn_{0.16}Cd_{0.84}Te/CdTe$ superlattices respectively.

The spectra obtained from 330Å period $Zn_{0.08}Cd_{0.92}Te/CdTe$ superlattices with (Zn,Cd)Te/CdTe layer thickness ratio of 1:1 and total superlattice thickness of 0.8µm grown on InSb(100) and CdTe(100) substrates are shown in Fig. 3. The spectra are similar, with the central peaks lying at a value corresponding with an average out-of-plane lattice parameter for the superlattice equal to that of $Zn_{0.04}Cd_{0.96}Te$. The $n=0$, Cu $K\alpha_1$ peak present as a shoulder on the InSb(400) $K\alpha_2$ peak, is not resolved for the superlattice grown on CdTe. The spectra obtained from an identical structure grown on GaAs(100) is shown in Fig. 4. The spectra are broad with no evidence of satellite peaks, indicating a highly disordered structure. Although excellent streaked RHEED patterns characteristic of a (2X1) reconstruction were observed during growth, a high density of dislocations was evident in cross-sectional transmission electron micrographs¹⁵ of similar samples grown in our laboratory. This is almost certainly a result of the 14% lattice mismatch between the (Zn,Cd)Te and GaAs.

Fig.5(a) and (b) show the spectra obtained for $Zn_{0.16}Cd_{0.84}Te/CdTe$ superlattices grown on InSb(100) and CdTe(100) substrates respectively with a total thickness of 1.6µm. The $Zn_{0.16}Cd_{0.84}Te:CdTe$ layer thickness ratio was 1:3 with a total period thickness of 330Å (85Å/250Å). The superlattice grown on InSb shows multiple satellite peaks from $n=-1$ to $n=+5$ with clear resolution of the Cu $K\alpha_1$ and $K\alpha_2$ lines.

indicative of a high structural quality. The (Zn,Cd)Te/CdTe 1:3 layer thickness ratio results in the satellite peak intensity asymmetry about $n=0$ as a result of the summed modulation of the two envelope functions arising from the (Zn,Cd)Te and CdTe layers. This envelope is broadened as a result of the thin 85Å (Zn,Cd)Te layer, allowing us to observe the higher order(+) satellite peaks. The superlattice grown on CdTe has a very poor diffraction spectrum, with ill defined and low intensity satellite peaks indicating a poor superlattice structure. This result was confirmed by repeating the superlattice growth on another CdTe substrate. An identical spectrum was obtained to that shown in Fig. 5(b).

To determine if the superlattice structures were strained, the in-plane lattice parameter of the 330Å $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ superlattice grown on InSb(100) was obtained by recording from the (220) and (440) reflections. This in-plane parameter was found to match that of the InSb substrate, indicating that this superlattice is strained and commensurate with the InSb(100) substrate.

DISCUSSION

The superlattices grown on InSb(100) are of a high structural quality as evident from the resolution of the Cu $K\alpha_1$ and $K\alpha_2$ lines, with no detectable change in the quality as the period thickness is decreased from 330Å to 85Å. There is no indication of structural differences for different superlattice thicknesses (0.8µm and 1.6µm). Satellite spacing and intensity for these samples are in good qualitative agreement with kinematic theory. Comparison of the 330Å period $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ and $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ samples grown on InSb(100) indicates very little difference in superlattice quality. There is no evidence that increased Zn content, and thus strain, is detrimental to the superlattice quality. There is very good agreement between the calculated and measured shift in the relative position of the satellite peaks due to the increase in the average Zn composition of the superlattice. Comparison of superlattice

growth on CdTe(100) and InSb(100) substrates indicates that superlattices grown on CdTe are inferior to those grown on InSb, probably a result of the higher defect density in CdTe substrates, although non-optimum preparation of the CdTe substrates cannot be ruled out. We were not able to grow thin ($<1.6\mu\text{m}$) high quality $(\text{Zn,Cd})\text{Te}/\text{CdTe}$ superlattices ($x<0.16$) on GaAs(100) substrates.

Excellent quality $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ superlattices can be grown on InSb(100). Determination of the in plane lattice parameter for a $85\text{\AA}/250\text{\AA}$ $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ superlattice indicates that this superlattice is strained and commensurate with the InSb(100) substrate. Since this sample has the maximum misfit and thickest layers (250\AA) of the samples studied, we may conjecture that all superlattices in this study have individual layer thicknesses below the critical thickness and are therefore strained, although not necessarily commensurate with the respective substrate. Because the $85\text{\AA}/250\text{\AA}$ $\text{Zn}_{0.16}\text{Cd}_{0.84}\text{Te}/\text{CdTe}$ superlattice is coherently strained the entire superlattice should meet the critical thickness criteria appropriate to an alloy of the same thickness and volume averaged composition, $\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}$ in this instance. Considering the mismatch for the $\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}/\text{InSb}$ system we find that this superlattice thickness ($1.6\mu\text{m}$) exceeds the Van der Merwe^{7,10} and Matthews and Blakeslee^{8,10} critical thickness models, but lies below that predicted by People and Bean^{9,10}.

CONCLUSION

Superlattices consisting of $\text{Zn}_x\text{Cd}_{1-x}\text{Te}$ ($x<0.16$) and CdTe have been grown by molecular beam epitaxy on CdTe(100), InSb(100) and GaAs(100) substrates. Multiple satellite peaks in X-ray diffraction spectra indicate that these superlattices can be grown with a high structural quality. We find that the quality is independent of the

superlattice period for period thicknesses between 85Å and 330Å, but sensitive to the substrate.

While measurement of the in-plane lattice parameter indicates that these superlattices are strained, it is uncertain if any are free standing, and thus lattice matched to $\text{Hg}_{0.8}\text{Cd}_{0.2}\text{Te}$. For superlattices grown on InSb it appears that the total superlattice thickness must be greater than $1.6\mu\text{m}$ if the superlattice is to be free standing.

Further studies by TEM and X-ray topography will be necessary to determine whether these superlattices are successful in bending and blocking dislocations and if these structures are superior to single film $\text{Zn}_{0.04}\text{Cd}_{0.96}\text{Te}$ epilayers for (Hg,Cd)Te epilayer growth.

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FIGURE CAPTIONS

Figure 1

Diffraction scans from the (400) reflection for $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ superlattices grown on $\text{InSb}(100)$ substrates with nominal period thicknesses of 85Å, 165Å and 220Å. The $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}:\text{CdTe}$ layer thickness ratio was 1:1 with a total superlattice thickness of 1.6µm.

Figure 2

Diffraction scans from (400) reflection for (a) 50 period 165Å/165Å $\text{Zn}_{0.18}\text{Cd}_{0.82}\text{Te}/\text{CdTe}$ superlattice (b) 50 period 165Å/165Å $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ superlattice.

Figure 3

Diffraction scans from (400) reflection of a 25 period 160Å/160Å $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ superlattice grown on (a) $\text{CdTe}(100)$, (b) $\text{InSb}(100)$.

Figure 4

Diffraction scan from (400) reflection of a 50 period 165Å/165Å $\text{Zn}_{0.08}\text{Cd}_{0.92}\text{Te}/\text{CdTe}$ superlattice grown on $\text{GaAs}(100)$.

Figure 5

Diffraction scan from (400) reflection of a 50 period 85Å/250Å $\text{Zn}_{0.18}\text{Cd}_{0.82}\text{Te}/\text{CdTe}$ superlattice grown on (a) $\text{InSb}(100)$, (b) $\text{CdTe}(100)$

